भारतीय मानक Indian Standard

साबुन स्टॉक — विशिष्टि

IS 12031: 2022

(पहला पुनरीक्षण)

Soap Stock — Specification

(First Revision)

ICS 71.100.40

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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Soaps and Other Surface Active Agents Sectional Committee had been approved by the Chemical Division Council.

Soap stock is produced during the refining of oils by alkali. The free fatty acids present in the starting oils get converted into soap which is soluble in water, but soluble to a very limited extent in the oil. The aqueous soap layer is separated by gravity or centrifuge. It contains three major components: (a) soap produced by reaction with fatty acid, (b) some emulsified neutral oil, and (c) water containing electrolytes like sodium chloride which are often added to reduce viscosity and emulsion formation. Depending on the method of refining, the soap stock will have different levels of moisture.

Soap stock, because of its expensive fatty matter content, cannot be wasted and is utilised for soap making. Because of the impurities removed from the oil during refining, the soap stock fatty matter is usually inferior to the starting oil. Being an aqueous medium with heavy organic loading, it is subjected to microbial action and degradation, which further degrades the quality of fatty matter contained therein. Both from the point of view of transportation and to ensure stability, it is necessary to remove the extra water as early as possible. This is achieved by converting it into acid oil.

This standard was originally published in 1986. This revision has been taken up in order to bring out the standard in latest style and format of the Indian Standards. The relevant clauses have been added and the references have been updated. Also in this revision, a requirement for maximum phosphorus limit content is added and a test method for the determination of phosphorus content is also added.

The composition of the Committee responsible for the formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2: 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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Indian Standard

SOAP STOCK — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for soap stock.

2 REFERENCES

The Indian standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Indian standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No</i> 286 : 2018	Title Methods of sampling and test for soaps (third revision)
548 (Part 1/Sec 1): 2021	Methods of sampling and test for oils and fats: Part 1 Sampling, physical and chemical tests, Section 1 Sampling
548 (Part 1/Sec 2): 2021	Methods of sampling and test for oils and fats: Part 1 Sampling, physical and chemical tests, Section 2 Physical and chemical tests

548 (Part	Methods of sampling and test for		
3/Sec 6):	oils and fats: Part 3 Advanced		
2021/ISO	instrumental methods, Section 6		
10540-1:	Determination of phosphorus		
2003	content by colorimetric method		
1070 : 1992	Reagent grade water- Specification (third revision)		
2711 : 1979	Specification for direct reading pH meter (second revision)		
11476 : 1985	Glossary of terms relating to oils and fats		

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 11476 shall apply.

4 REQUIREMENTS

4.1 Description

The material shall be an aqueous medium with heavy organic loading.

4.2 The material shall also conform to the requirements given in Table 1, when tested according to methods prescribed in co1 (4) of Table 1.

Table 1 Requirements for Soap Stock

(*Clause* 4.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Total fatty matter (TFM), percent by mass, <i>Min</i>	20	IS 286
ii)	pH of 1 percent solution, Min	7.0	A-2 of Annex A
iii)	Unsaponifiable matter, percent by mass on TFM, <i>Max</i>	5.0	IS 548 (Part 1/Sec 2)
iv)	Oxidized fatty acid, percent by mass on TFM, Max	5.0	A-3 of Annex A
v)	Phosphorus content, ppm, Max	150	IS 548 (Part 3/Sec 6)

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable containers or transported through tankers or as agreed to between the purchaser and the supplier.

5.2 Marking

- **5.2.1** The material, if packed in containers, shall be securely closed and marked with the following information:
 - a) Name of the material;
 - b) Manufacturer's name and recognized trademark, if any;
 - c) Net mass of the material;
 - d) Batch number or lot number in code or otherwise; and
 - e) Month and year of manufacture.

5.2.2 The containers shall also, in addition, be legibly and indelibly marked with the information required under the Standards of Weights and Measures (Package Commodities) Rules, 1977.

5.2.3 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 548 (Part 1/Sec 1).

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ANNEX A

[Table 1, Sl No. ii) and iv)]

METHODS OF TEST FOR SOAP STOCK

A-I QUALITY OF REAGENTS

Unless stated otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF pH

A-2.1 General

The pH determination should be made in an acid free atmosphere.

A-2.2 Apparatus

A-2.2.1 *pH Meter*

Any standard electro-metric instrument, equipped with a low sodium error glass electrode. The instrument shall be calibrated and standardized with standard buffer solutions (*see A-2.3.2*) before use (*see IS 2711*).

A-2.2.2 Volumetric Flask — 1 000 ml Capacity

A-2.2.3 Beaker — 100 ml Capacity

A-2.3 Reagents

A-2.3.1 Distilled Water

Distilled water shall be boiled thoroughly or purged with carbon dioxide-free air to remove carbon dioxide, and shall be protected with soda lime or soda asbestos while cooling and in storage. The pH of this water shall be between 6.2 and 7.2 at 27 °C. The residue on evaporation when heated at 105 °C for one hour shall not exceed 0.5 mg per litre.

A-2.3.2 Standard Buffer Solution

Any two suitable buffer solutions within the pH range of 9 to 11 at 27 °C for calibrating the pH meter.

A-2.4 Procedure

A-2.4.1 Weigh 10 g \pm 0.001 g of the material and transfer to a 1 litre volumetric flask. Partially fill the flask with distilled water and agitate until the sample is completely dissolved. Adjust the temperature of the solution and the distilled water to 27 °C \pm 0.5 °C, and fill to the calibration mark with the distilled water. Stopper the flask, mix thoroughly, and allow the solution to stand at a temperature of 27 °C for 2 hours prior to measuring the *p*H. Measure the *p*H of the solution using a glass electrode.

A-3 DETERMINATION OF OXIDIZED FATTY ACID

A-3.0 General

A known quantity of the material is saponified with alcoholic potash and the soap formed is treated with mineral acid to release the fatty acids. The petroleum ether insoluble but ethyl ether soluble matter, which is termed as oxidized fatty acid, is determined by evaporation of the ethyl extract of the residual aqueous layer left behind after the petroleum ether extraction.

A-3.1 Apparatus

A-3.1.1 Stoppered Glass Cylinder — 250 ml Capacity.

A-3.1.2 Separating Funnel — 500 ml Capacity.

A-3.1.3 Flasks — 250 ml Capacity, Flat Bottom; and 300 ml Capacity, Conical.

A-3.2 Reagents

A-3.2.1 Alcoholic Potassium Hydroxide Solution

Dissolve 50 g of potassium hydroxide in 1 litre of 95 percent ethyl alcohol.

A-3.2.2 *Dilute Hydrochloric Acid* — 1:1 By Volume.

A-3.2.3 Methyl Orange Indicator

Dissolve 0.1 g of methyl orange indicator in 100 ml of water.

A-3.2.4 Petroleum Ether — 60°C/80°C Distillation Range.

A-3.2.5 Ethyl Ether

A-3.3 Procedure

A-3.3.1 Weigh accurately 3 g to 5 g of the fatty matter into a 300 ml conical flask. Add 50 ml of alcoholic potash, cover with an inverted funnel and heat on a water-bath to saponify. Agitate frequently and heat for at least 30 minutes or until saponification is complete.

A-3.3.2 Remove the watch-glass and continue heating on a water-bath with occasional agitation to evaporate the alcohol. To avoid oxidation, do not evaporate beyond a pasty mass. If necessary, add a small amount of water when most of the alcohol has evaporated.

- **A-3.3.3** Add 100 ml of distilled water and heat until the soap has completely dissolved. Wash the contents into a glass-stoppered cylinder with hot distilled water, taking care not to exceed a total volume of 130 ml in the cylinder.
- **A-3.3.4** Add 3 drops to 5 drops of indicator and neutralize with hydrochloric acid to the pink methyl orange end point. Then add 1 ml of excess acid. Rotate the cylinder gently to mix the contents.
- **A-3.3.5** Cool to at least 35 °C and add 125 ml of petroleum ether. The fatty acids need not have cleared completely before adding the ether. Stopper the cylinder, shake gently, and allow to stand until the petroleum ether layer separates.
- A-3.3.6 Siphon the petroleum ether layer into a 500 ml separating funnel, making sure that as little as possible of the insoluble matter which gathers at the ether-water interface is carried over into the separating funnel. If any appreciable amount of insoluble matter does siphon over into the separating funnel, it will usually settle to the bottom and shall be drained back into the extraction cylinder. Make at least 4 more similar extractions using 25 ml to 30 ml of petroleum ether, shaking the cylinder vigorously for 30 seconds with each extraction. Extractions shall be continued until the petroleum ether layer is practically colourless.
- A-3.3.7 To the acid water remaining in the extraction cylinder add 25 ml to 30 ml of ethyl ether, stopper, shake gently and allow to stand until the ether layer separates. Siphon the ethyl ether layer through a filter paper into a tared 250 ml flat bottom flask which has been dried and cooled in a desiccator. Make at least 4 more similar extractions using 25 ml to 30 ml of ethyl ether each time, and shake the cylinder vigorously for 30 seconds with each extraction. The last ethyl ether extract shall be practically colourless.
- **A-3.3.8** Filter all extracts through the same filter paper and finally wash this filter paper thoroughly with ethyl ether to recover all the oxidized acids.
- **A-3.3.9** Evaporate the ethyl ether extracts on a water-bath under a gentle stream of clean dry air. Finally, dry the oxidized fatty acids in an air oven at $105~^{\circ}\text{C} \pm 2~^{\circ}\text{C}$ for 30 minutes. Cool in a desiccator to room temperature and weigh. Repeat until constant mass, that is, to within 0.1 percent between successive weighings, is obtained.

A-3.4 Reporting

Report the oxidized fatty acids as a percentage of the material taken for the test.

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ANNEX B

(Foreword)

COMMITTEE COMPOSITION

Soaps and Other Surface Active Agents Sectional Committee, CHD 25

Organization Representative(s)

Harcourt Butler Technological University, Kanpur PROF RAKESH K. TRIVEDI (Chairperson)

Central Drugs Standard Control Organization, New Delhi Shri Sudipta Dey

Central Pollution Control Board, New Delhi Shri Dinabandhu Gouda

SHRI A. SUDHAKAR

Consumer Guidance Society of India, Mumbai DR SITARAM DIXIT

SHRI B. V. DESAI

Consumer Voice, New Delhi Shri M. A. U. Khan

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Procter & Gamble India, Mumbai SHRI GIRISH PARHATE

SHRI SIVAKUMAR THANIGACHALAM

Reckitt Benckiser India Limited, Mumbai SHRI RUCHIR SHAH

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BIS Directorate General SHRI A. K. LAL, SCIENTIST 'F' AND HEAD (CHD)

[REPRESENTING DIRECTOR GENERAL (Ex-officio)]

Member Secretary Puja Priya SCIENTIST 'C' (CHD), BIS This Pade has been Intentionally left blank

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